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Atomizer for Flame Spectrophotometry. C. O. Willits and J. A. Connelly, Eastern Regional Research Laboratory, Philadelphia 18, Pa.

IN THE analysis of agricultural products, it is often necessary to determine trace amounts of sodium, potassium, magnesium, and calcium. Flame photometry is particularly well suited to the determination of these elements, but because of the limited amount of sample, an atomizer is needed that uses a minimum of sample and meets precision requirements. An atomizer of the reflux type, which uses only 0.3 to 0.4 ml. of sample solution per minute, was designed for this purpose. In addition to meeting precision needs, it proved to be free of changing nozzle characteristics due to salt encrustation, and a minimum of time and manipulation was required for cleaning it.

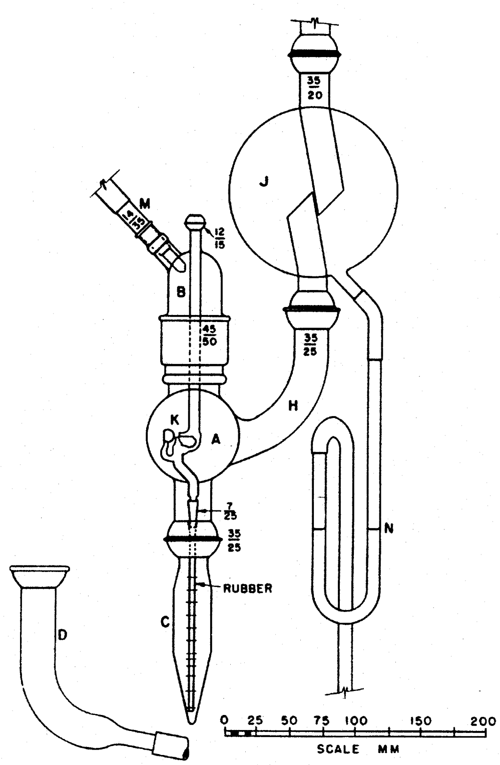

Figure 1. Reflux-Type Glass Atomizer

The all-glass atomizer (Figure 1) consists of an atomizer chamber, *A*, a sphere 70 mm. in diameter. An outer 45/50 standard-taper joint for connecting the cap assembly is sealed at the top, *B*, and sealed to the bottom is the ball of a 35/25 ball-and-socket joint for attaching either the sample tube, *C*, or washing tube, *D*. To obtain greatest flexibility, ball joints were used throughout except for the cap assembly. The aerosol tube, *H*, 28 mm. in inside diameter, sealed to the side of the chamber, leads to the spray separator, *J*, a sphere 120 mm. in diameter containing overlapping inlet and outlet tubes and a drain tube with trap, *N*. The outlet tube of the separator is connected to the tube leading to the burner. Attached to *B*, made from the inner part of a 45/50 standard-taper joint, is the washing assembly, *M*, and the tube supporting the atomizer assembly, *K*. *K* is kept in position by the rigid tapered joint connecting the cap and atomizer chamber.

M consists of a nozzle made from the inner joint of a 14/35 standard-taper joint, which is held in position by insertion in a 14/20 outer joint sealed to the cap at a 45° angle. This arrangement permits a 180° rotation of the nozzle in the 14/20 joint, to wash all surfaces of the cap and atomizer chamber. The detachable and interchangeable sample tubes, *C*, are made from graduated 50-ml. conical centrifuge tubes, which are attached to the lower end of the atomizer chamber with a 35/25 socket joint. The interchange of sample tubes is facilitated and danger of breakage of the rigid atomizer assembly is minimized by use of a rubber tube for aspirating the sample from the sample tube.

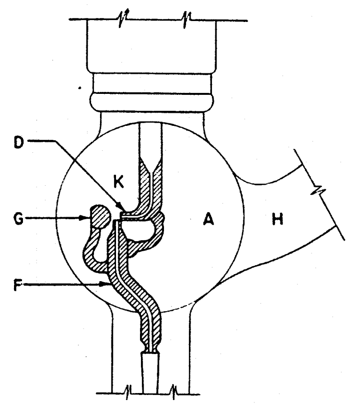

Figure 2. Atomizer Assembly

The atomizer assembly (*K*, Figure 2) consists of an air jet, *D* with a nozzle 5 mm. long made of 2-mm. heavy-walled capillary tubing; the nozzle is directed to the side of the chamber opposite that of the aerosol tube, *H*. Attached to the air jet, with a glass bridge, is the liquid nozzle mounted at right angles to the air nozzle. The liquid nozzle on the aspirator tube, *F*, is 5 mm. long, and it also is made from 2-mm. heavy-walled capillary tubing. Sealed to the liquid nozzle directly in front of the air jet is a glass ball, *G*. The lower end of the glass aspirator tube has an inner 7/25 standard-taper joint, to which can be attached either a rubber or glass extension of the tube. Glass is used if the solvent attacks rubber.

Operation. In flame spectrophotometry, the sample is excited by atomizing it into a flame. The stream of air from the air nozzle, operating at 5 to 15 pounds per square inch, aspirates the sample from the sample tube. As the sample leaves the nozzle, it comes in contact with the high-velocity flow of air and is dispersed into droplets. The droplets are further reduced in size by impinging them on the glass ball. The large droplets in the spray are carried to the chamber wall, opposite opening *H*, where they coalesce and run back to the sample tube. Any remaining large droplets are removed when the fog passes through the separator. The essentially "dry" aerosol, as demonstrated by its nonfogging of a mirror, is led to the flame. This aerosol is stable and can, therefore, be piped for distances up to 20 feet, providing the conduit is of uniformly large diameter and free of sharp bends. In this type of atomizer, the air used for atomization causes evaporation of the sample solution, bringing about a continuous concentration of the sample, which in turn changes the flame intensity. To prevent concentration of the sample, the atomizer is operated with air saturated with water. When a solvent other than water is used, the air is saturated with that solvent.

Washing the Atomizer. Provision has been made for easy washing of the atomizer while it is in operating position. This makes it unnecessary to dismantle the atomizer between analyses.

The air is kept on, and the sample tube is replaced by the drain tube (Figure 1, *D*), which is connected to a sink with rubber tubing. This rubber drain tube is closed with a pinchcock, and rinse water with a head of 5 feet is flushed through the nozzle in the atomizer cap. As the water rises in the drain tube, it is aspirated through the nozzle, rinsing it; flood rinsing of the apparatus is continued until the water overflows into the separator. The flow of rinse water is then stopped, the pinchcock on the drain tube is removed, and the system is drained. Water in the separator is automatically drained through the trap. Rinsing is completed in 15 to 20 seconds.

Experimental. A solution containing 15 p.p.m. of potassium chloride was atomized, and the aerosol was led into a propane-oxygen flame to measure the constancy of intensity of the radiation. The intensity values were constant during a 15-minute atomization period. This eliminates the need for establishing empirically the time at which observations must be taken.

The atomizer requires a minimum of manipulation and has given reproducible results. The excited aerosol emits a constant radiation, as demonstrated by the steadiness of the galvanometer needle when the instrument is operated at its highest sensitivity. The stability of the aerosol is further demonstrated by the distance which it can be "piped" to the flame. The latter property has made possible the safe use of flammable test solutions, because atomizer and sample tube assembly may be located at a safe distance from the flame.

The authors acknowledge the assistance given by Harry John of this laboratory, in the construction of the apparatus.

PRESENTED at the Meeting-in-Miniature, Philadelphia Section, AMERICAN CHEMICAL SOCIETY, Philadelphia, Pa., January 1951.